Supporting Information

Robotic Lepidoptery: Structural Characterization of (mostly) Unexpected Palladium Complexes Obtained from High-Throughput Catalyst Screening

John E. Bercaw, Michael W. Day, Suzanne R. Golisz, Nilay Hazari, Lawrence M. Henling, Jay A. Labinger,* Susan J. Schofer,[†] Scott Virgil*

Arnold and Mabel Beckman Laboratories of Chemical Synthesis, California Institute of Technology, Pasadena, California 91125 and [†]Symyx Technologies, Inc., 1263 East Arques Avenue, Sunnyvale, CA 94085

(a) Selected ^{1}H NMRs

(b) Crystal and refinement data of **5m**

(c) Selected bond distances of **6**

(a) Selected ^{1}H NMRs

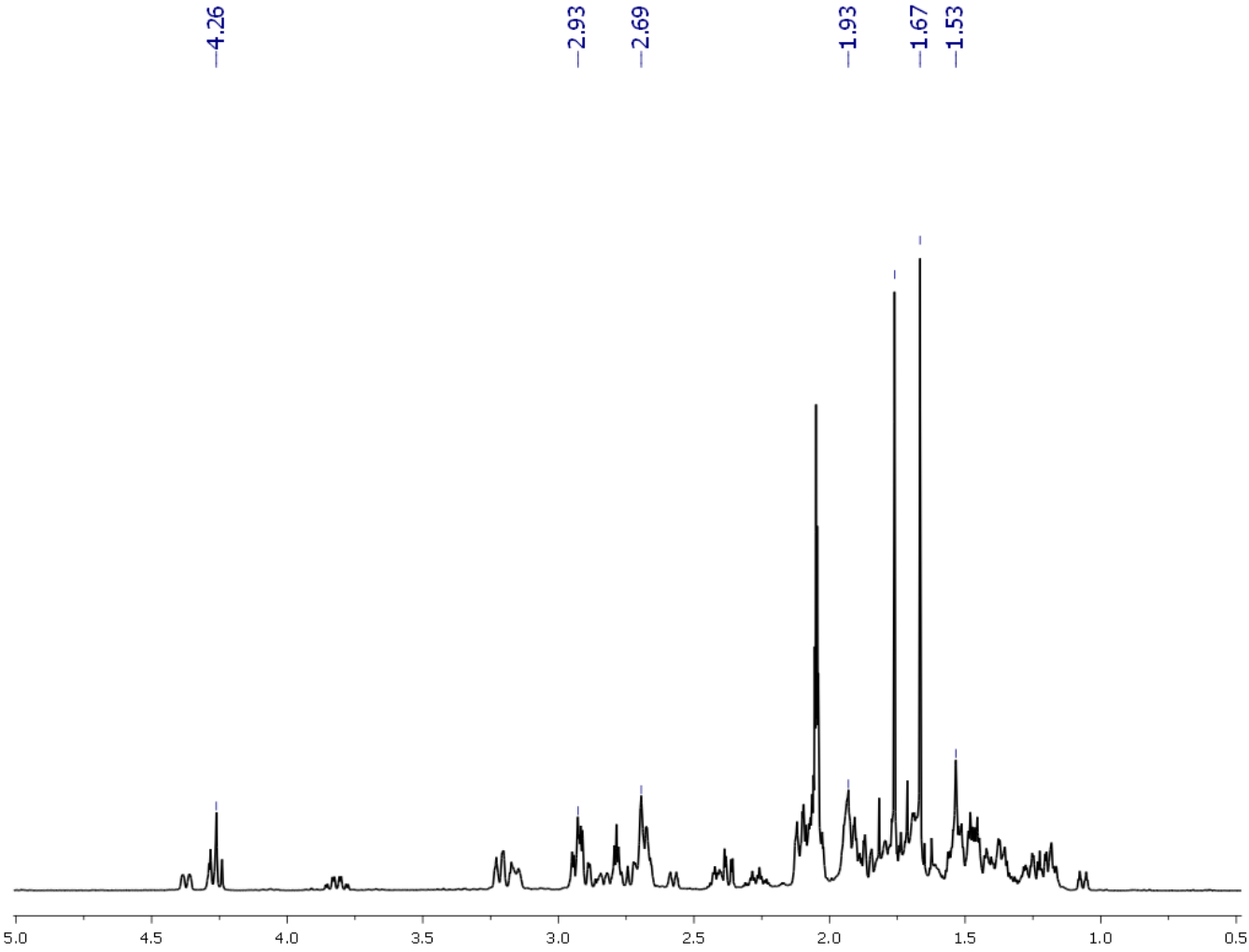

Figure S1. ^{1}H NMR spectrum of (sparteinyl)Pd(μ-OC(CH$_3$)O)$_2$Pd(μ-OC(CH$_3$)O)$_2$Pd(sparteinyl) (**4**).

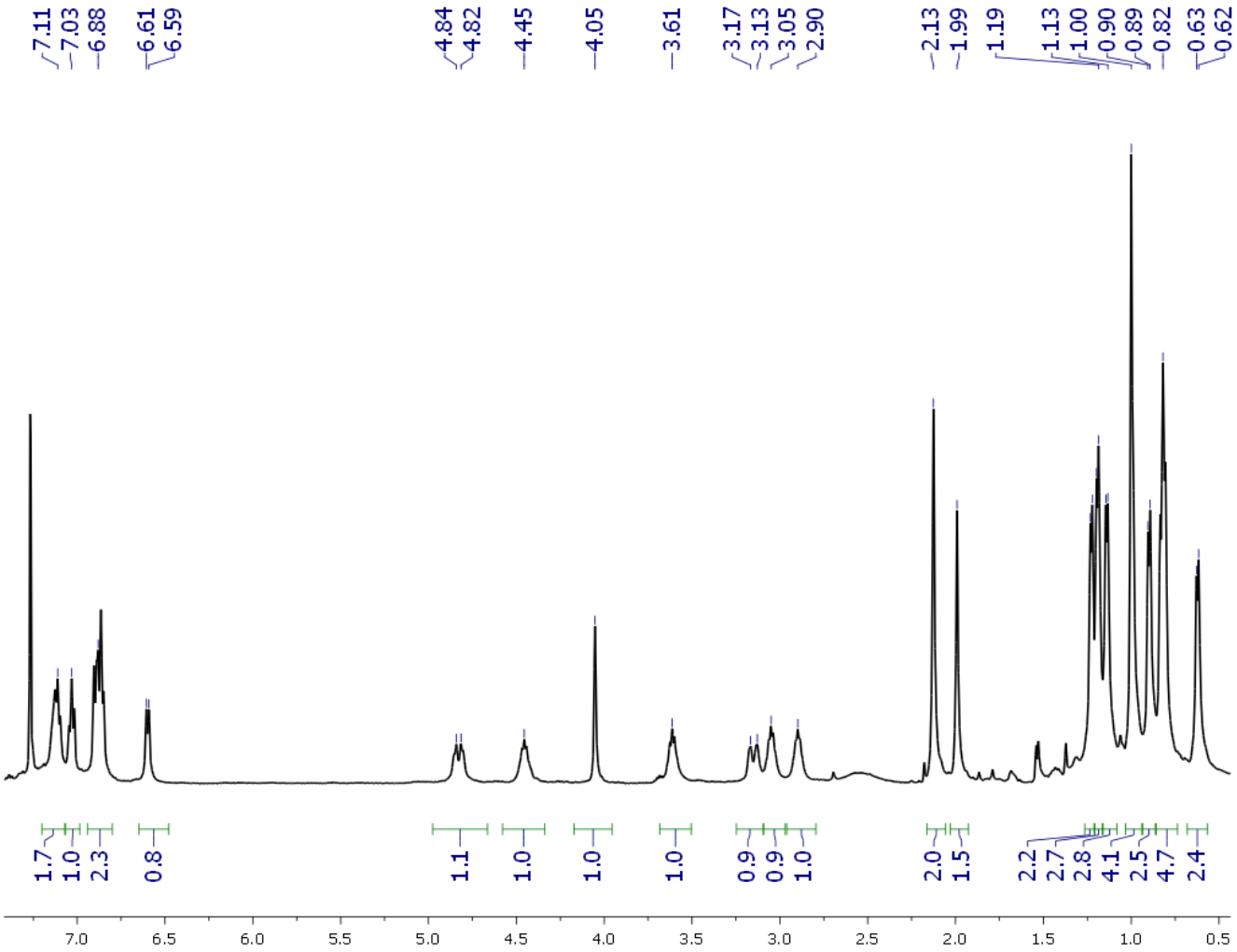

Figure S2. ^{1}H NMR spectrum of $[(\text{diimine})(Pd)(OAc)_2]_{10}$ (**6**).

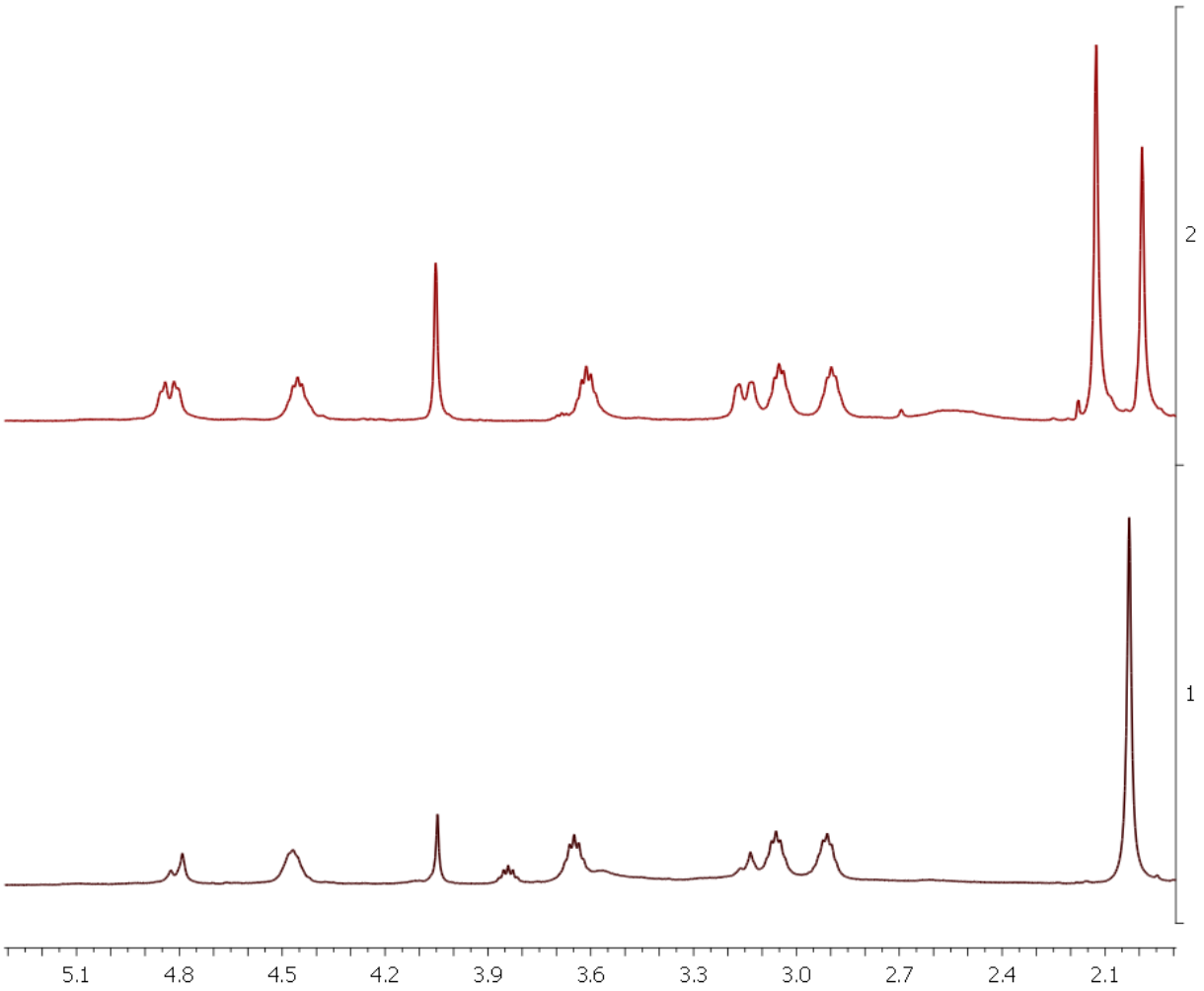

Figure S3. ^{1}H NMR spectra of [(diimine)(Pd)(OAc)$_2$]$_{10}$ (**6**) and [(diimine)(Pd)(OAc)$_2$]$_{10}$-d_n (**6**-d_n). The top trace is the protio complex and the bottom trace is the deutero complex. The patterns for the signals around δ 4.8 and 3.15 in the bottom trace both arise from a overlay of peaks from the CH_2 (doublet) and CHD (singlet, shifted slightly upfield from the midpoint of the doublet) isotopologs.

(b) Crystal and refinement data of **5m**

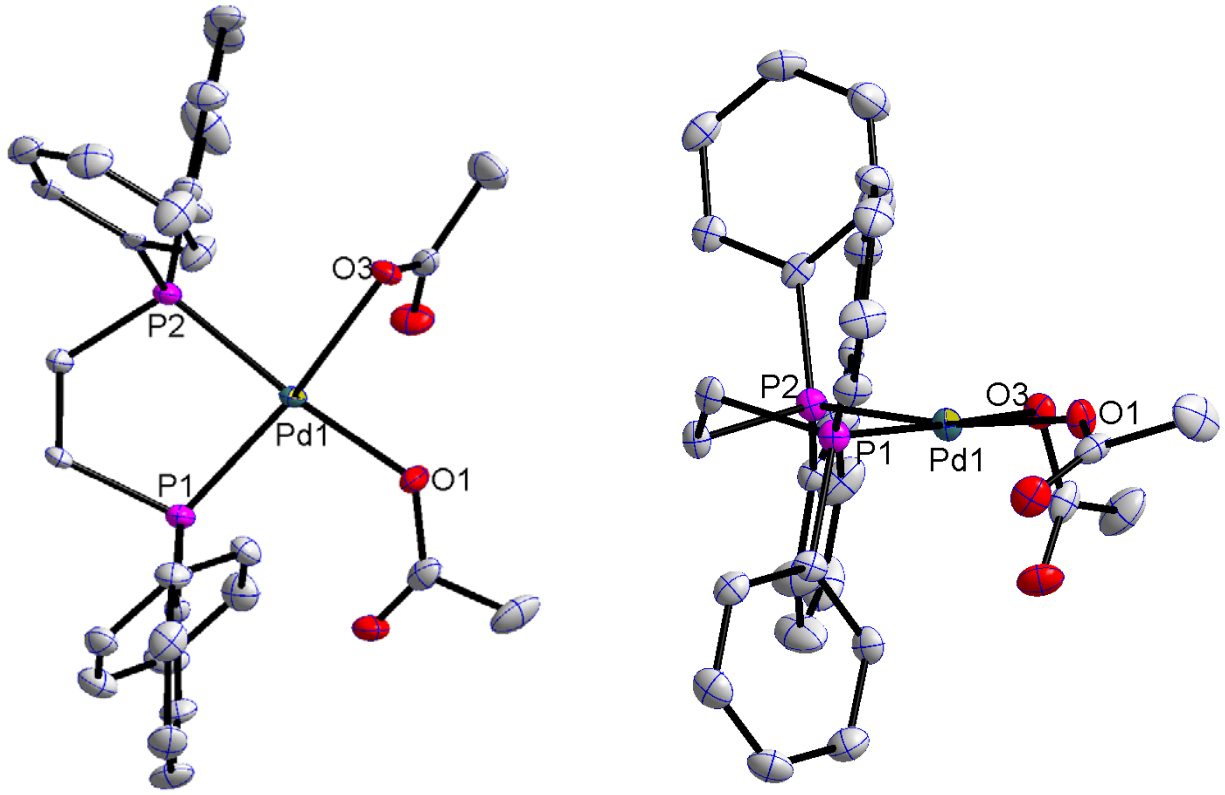

Figure S4. The solid state structure of **5m**. Hydrogens have been omitted for clarity. Selected bond distances of **5m** (Å): Pd1-O1 2.060(2), Pd1-O3 2.107(2), Pd1-P1 2.229(9), Pd1-P2 2.217(9).

Table S1. Crystal and Refinement Data for Complex **5m**.

	5m
Empirical formula	$C_{30}H_{30}O_4P_2Pd \bullet 0.5(C_3H_6O)$
Formula weight	651.92
Temperature (K)	100
a (Å)	11.7614 (10)
b (Å)	19.1959(18)
c (Å)	14.3280(13)
α (deg)	90
β (deg)	113.600(4)
γ (deg)	90

Volume (Å^3)	2964.3(5)
Z	4
Crystal system	Monoclinic
Space group	$P2_1/c$ (#14)
d_{calc} (Mg/m^3)	1.461
θ range (deg)	1.88 to 30.07
μ (mm^{-1})	0.770
Abs. correction	Semi-empirical from equivalents
GOF	1.46
R_1,[a] wR_2[b] $[I>2\sigma(I)]$	0.065, 0.064

[a] $R_1 = \Sigma||F_o| - |F_c||/\Sigma|F_o|$. [b] $wR_2 = \Sigma[w(F_o^2 - F_c^2)^2]/\Sigma[w(F_o^2)^2]^{1/2}$.

(c) Selected bond distances of **6**

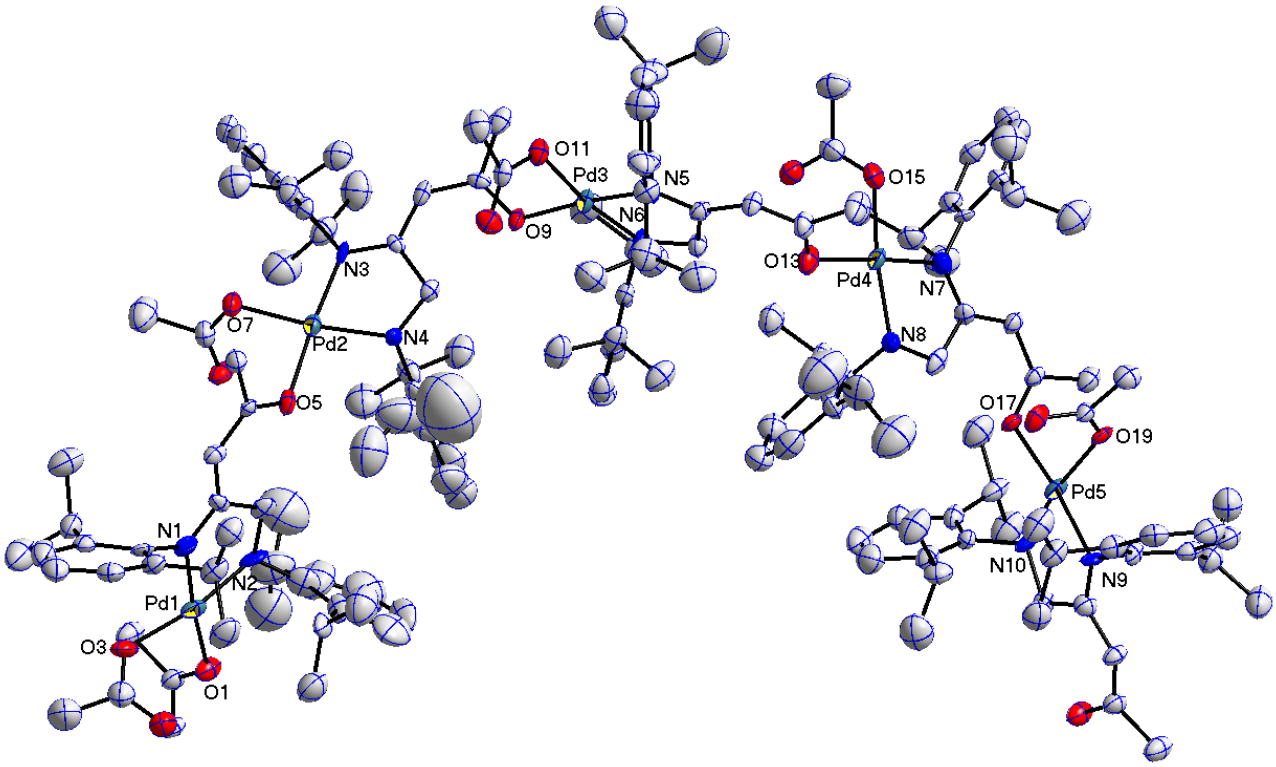

Figure S5. The asymmetric unit of **6**.

Table S2. Selected bond lengths (Å) for **6**.

Pd(1)-O(3)	1.985(8)
Pd(1)-N(1)	1.997(9)
Pd(1)-N(2)	2.018(10)
Pd(1)-O(1)	2.020(7)
Pd(2)-N(3)	1.959(9)
Pd(2)-O(5)	2.017(7)
Pd(2)-O(7)	2.025(8)
Pd(2)-N(4)	2.042(9)
Pd(3)-N(5)	1.977(9)
Pd(3)-O(9)	2.007(7)
Pd(3)-O(11)	2.019(7)
Pd(3)-N(6)	2.060(8)

Pd(4)-N(7)	1.977(9)
Pd(4)-O(15)	1.983(7)
Pd(4)-O(13)	2.027(8)
Pd(4)-N(8)	2.037(8)
Pd(5)-O(17)	2.021(7)
Pd(5)-N(9)	2.013(8)
Pd(5)-N(10)	2.025(9)
Pd(5)-O(19)	2.020(7)